ORTEC®

Guidelines for Low Level Gamma Spectrometry – Air Filters, Water, and Soils

Part One – Gamma Spectroscopy Systems

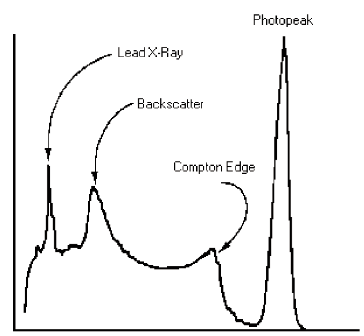
This document addresses effective techniques for analysis of environmental samples by gamma-spectrometry with the focus on appropriate techniques for analyzing air filters, water samples, and soils. A thorough examination of the topic is beyond the scope of this text. Rather, a brief description of a typical gamma spectrometry system is provided. The description highlights some common (yet critical) challenges that face the gamma spectroscopist and the ORTEC solutions to these challenges. References to published methods and research are provided for the reader to develop the final techniques.[1-10]

The American National Standards Institute document "American National Standard for Calibration and Use of Germanium Sprectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides," ANSI N42.14-1999 is referred to often in this summary.[1] The reader is encouraged to purchase a copy of this useful document.

Gamma Ray Measurement Systems

Typical Systems

A typical gamma spectrometry system consists of a Germanium (Ge) detector, liquid nitrogen or mechanical cooling system, preamplifier, detector bias supply, linear amplifier, analog-to-digital converter (ADC), multichannel storage of the spectrum, and data readout devices.[1] The detector is often housed within a shield to reduce the background caused by sources other than the sample. The shield is constructed of a dense material (such as lead) that will absorb a large portion of background gamma rays. The shielding is usually crafted in such a way as to minimize backscattering. The lead shielding material is usually graded with a two part thin metal shield such as tin and copper to reduce the effects of x-rays generated by the interaction of ambient photons with the lead. The sample is positioned within the shield at some distance from the detector. The distance will depend on a number of parameters, such as expected count rate and geometry of the sample container.



Using this hypothetical system, photons emitted from the sample interact with the Ge crystal to produce a pulse. The amplitude (or height) of the pulse is proportional to the energy of the photon absorbed by the Ge. Each pulse is amplified (or magnified), shaped, and sorted according to pulse height to produce a histogram (counts per unit energy) of the incident photons. This histogram is called a spectrum. As the counts accumulate, peaks develop that can be identified by energy and thus the nuclide identities of the spectrum are also identified — assuming that the system has been calibrated. In general, the goal of the gamma spectroscopist is to derive nuclide-specific gamma emission rates of the sample (in activity units, such as Becquerels [Bq] or decays per second) from the spectral data.

Despite the apparent simplicity with which gamma-ray measurements are made (little sample preparation is required), there are a number of correction factors to the raw counting data that must be considered:

- The loss of pulses due to pulse pile up (at high count rates).
- Coincidence summing (both random and cascading).
- The decay of the source during counting.
- The decay of the source from some previous reference time.
- Attenuation of photons as a result of interactions with the sample.
- Emission rate (or yield) of the specific photon energy.

Gamma-Ray Spectrometer Location

Germanium detectors should be located in areas where stable, clean power is available. An Uninterupted Power Supply (UPS) is recommended. The ambient temperature should be relatively stable and between 16°C and 27°C. The power supply for all components should be isolated from other instrumentation and share a common ground,[1] i.e., the NIM bin or integrated spectrometer, computer, and mechanical cooler (if used) should all be plugged into the same electrical circuit.

Ideally, the conditions should be adjusted so that they are within the ambient conditions outlined in the manufacturer's specification testing document. This ensures that validation tests of the manufacturer's specifications are reproducible to the extent possible. In general, the manufacturer's testing of the spectral quality of a Ge detector will include test results for:

- Full Width at Half Maximum (FWHM), 122 keV ^{57}Co and 1332 keV ^{60}Co (5.9 keV ^{55}Fe for N-type detectors and 14.4 keV ^{57}Co may be included for thin window GEM detectors).

- Full Width at Tenth Maximum (FWTM) 1332 keV ^{60}Co.
- Peak to Compton Ratio 1332 keV.

The FWHM (or full width of the peak at one-half the peak maximum), the FWTM (or full width of the peak at one-tenth the peak maximum), and the Peak to Compton Ratio are measures of the quality of the spectrum created by the system. These quality measures are known as peak resolution, peak shape, and full energy peak efficiency, respectively. They are measures of the ability to resolve (or separate) full-energy peaks. The values obtained for these quality measures are a function of the total system (detector quality, detector size, electronic noise suppression, etc.).

Guidelines for testing procedures can be found in ANSI N42.14 and IEEE Std 325-1996. A brief summary of these procedures is outlined below.

Calibration of Germanium Detector Systems

Fundamentals

The calibration of the Ge system is critical to achieving accurate results.[1-2] Proper calibration ensures that gamma ray spectra are accurately interpreted in terms of energy and specific activity (activity per unit mass, e.g., Bq/gram). If computer analysis is to be enacted, it may be necessary to provide information about the spectral quality (resolution) in order for the analysis software to accurately analyze the spectrum.[2] There are three main tasks:

- Energy calibration.
- Peak width calibration (as a function of energy).
- Efficiency calibration (the relationship between the number of counts and the disintegration rate).

Each of these tasks is in principle simple to accomplish, but there are potential sources of error that must be taken into consideration:[1-2]

- Efficiency changes due to variable source matrix (density).
- Anomalous peak widths.
- Effect of source/detector distance.
- Effect of sample density.
- Pile up losses.
- True coincidence summing.
- Inaccurate decay corrections.
- Live time correction errors.

Gamma spectrometers must be calibrated using high quality standard spectra of appropriate geometry and source matrix for the sample to be measured. Standards may be obtained from a number of source manufacturers (or prepared in the lab). For traceability (or relation of results to a recognized standards organization), sources should be obtained from a manufacturer that maintains an acceptable Quality Assurance Program (QAP) and participates in a Measurement Assurance Program (MAP) with a recognized standards organization, such as the National Institute of Standards and Technology (NIST).[3] Other international standards organizations include: The National Physics Laboratory (NPL) in the United Kingdom and Physkalisch-Techniche Bundsenstalt (PTB) in Germany. It is recommended that the Quality Assurance Program of the source manufacturer be reviewed prior to the purchase of standards. In addition, in order for results to be considered reasonable, the nuclear data used in the calculations must be from a recognized standard source.[2]

Energy Calibration

Energy calibration is accomplished by measuring the spectrum of a source with known full-peak energies. The source may contain a single nuclide (e.g., Eu-152), or a mixture of gamma emitters such as the Mixed Gamma Standard, which contains Co-57, Sn-113, Hg-203, Cd-109, Ce-144, Cs-137, Co-60, and Y-88. It is critical that sources consist of known energy peaks that encompass the entire energy region over which the spectrometer is to be used.[2] The source need only be counted long enough to identify the peak energies in the spectrum. However, the statistics of subsequent measurements demand that sufficient counts be obtained in each energy peak to ensure that adequate uncertainty levels are achieved. ANSI N42.14 suggests that spectra should be collected to achieve a precision of less than 0.2 keV in peak position. The peak positions at the calibration energies are used to specify the mathematical relationship of the peak fitting functions of the calibration software. These mathematical relationships are stored for evaluating the peak energies of subsequent sample counts. If the Ge detector has adequate long-term stability, these relationships need only be checked each day using a calibration source.[1]

Peak Width Calibration

If a computer analysis is used to determine the specific activity of the sample, a peak width (and peak shape) calibration is necessary. The procedure is much the same as the energy calibration procedure and may be done simultaneously.[2]

The peak resolution, at high and low energy, should be measured and recorded on a regular basis. To be consistent, the number of counts collected in the regions to be monitored should be roughly the same for each measurement. ANSI N42.14 suggests a procedure that includes the following for determining the FWHM at 1332 keV:

1. Establish the settings of the Ge spectrometer system so that the gain is approximately 0.5 keV/channel for 4k channels and 0.25 keV/channel for 8k. This puts Co-60 in channels 2664 and 5328 respectively. This gain corresponds to placing the 1332 keV full-energy peak of Co-60 in channel 2664 and 5328 respectively
2. Center the source on the detector axis at a distance from the detector that establishes a counting rate of about 500 s^{-1}.
3. Acquire a spectrum with about 3000 counts in the peak channel for the gamma ray at 1332.50 keV. If possible, adjust the fine gain so there is an integer channel located within ±0.1 channels of the peak position, and re-acquire a spectrum.
   - Having an integer channel located at the mean channel of the peak simplifies the measurement of the energy resolution.
4. Perform an energy calibration using the 1173.24 keV and 1332.50 keV gamma rays (by inclusion of a Co-60 source, the gain may be measured more accurately). If the ADC zero was previously set, the intercept term should be close to zero, even at a different gain.
5. Determine the FWHM by the equation:

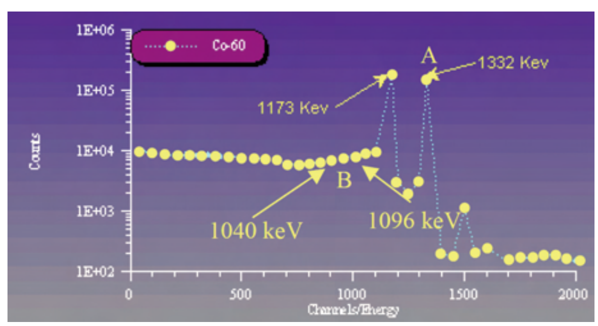
$$FWHM = 0.94^{*}\left(\frac{N}{Y}\right)$$

where

N = net peak area
Y = peak height

An alternative method commonly used to measure the FWHM is to subtract the baseline channel-by-channel from the gross counts in the peak, determine the number of channels (to within ±0.2 channels) above the half-height of the peak and convert the number of channels to energy.

Peak-to-Compton Ratio



The peak-to-Compton Ratio (P-to-C) is defined as the ratio of the number of counts in the biggest channel of the 1332.50 keV Co-60 energy peak to the average channel count in the Compton continuum between 1040 and 1096 keV in the same spectrum (Fig. 1). As such, the P-to-C is a spectral quality indicator that includes elements of both detector resolution and full-energy peak efficiency. The ratio is analogous to a signal to noise ratio.[2] One can easily see that the better the resolution, the larger the value of A and the greater the P-to-C will be. In the same way, it can be seen that the greater the detector efficiency at the full-energy peak, the greater the value of A. Larger detectors will have a greater probability of capturing all the gamma energy and will have more counts in the peak and fewer in the Compton continuum, increasing the P-to-C value from both ends.[2] The P-to-C can be checked with any Co-60 source as long as the source material is of sufficiently low Z material to ensure that interaction with the source material is minimized. For this reason, point sources in thin film (or tape) are generally used to check the P-to-C ratio of a given detector.

Review of Part One — Preview of Part Two

Part One of this text has summarized a number of common challenges that face todays gamma-spectroscopist. The ORTEC brand of instruments offers a complete spectroscopy solution for each of these common problems. In Part Two, the ORTEC solutions to these problems and challenges is summarized and recommendations are presented as guidance in choosing the best components to suit the analysis need.

Guidelines for Low Level Gamma Spectrometry – Air Filters, Water, and Soils

Part Two — Precise Solutions

Hardware Solutions — ORTEC Detectors and Electronics

High-Purity Germanium Detectors

High-resolution gamma-ray measurements for environmental sample analysis require the use of High Purity Germanium (HPGe) detectors. ORTEC has consistently produced a variety of high-quality HPGe detectors to match the analysis needs of the gamma-spectroscopy community. ORTEC's HPGe manufacturing facility is one of only three in the world that can produce spectroscopy grade germanium.

High-resolution gamma spectroscopy provides fast, accurate, non-destructive isotopic analysis of natural and anthropogenic radionuclides in environmental samples. Larger volume high-purity germanium detectors, with relative efficiencies over 100% and less than 2.0 keV FWHM resolution for the 1332-keV Co-60 peak, provide improved peak to background ratios which translate directly into better sensitivity (lower MDAs) for a given counting time.[4]

Sample-Counting Geometry Optimization

It is argued that germanium detector use and germanium crystal production has progressed to such a degree that the IEEE standard for specifying the performance of the detector is no longer adequate to predict the efficacy for a particular experimental situation.[5] More than ever, germanium detectors are being used for increasingly specialized applications with varying spectral, statistical, and detection limit requirements. The specification of the resolution, relative efficiency and peak-to-Compton ratio for a Co-60 point source at 25-cm distance does not predict adequately how a detector will perform at low energies and for other geometries. Thus, to select the best detector suited for an application, it is necessary to examine the variables that contribute to the overall effectiveness of the measurement. Keyser (1999)[6] provides examples of the efficiency, minimum detectable limit, throughput and resolution for a number of different source-detector configurations and crystal-preamplifier configurations. These source-detector configurations correspond to applications in the area of high-energy prompt gamma ray detection, filter paper samples for air monitoring, high-resolution, high-rate counting for safeguards and Marinelli samples in environmental counting. Selection of the right detector-sample geometry is critical for optimizing the experimental conditions for each sample type. This work suggests the following guidelines for selecting the detector best suited for an application:[6]

1. The more detector material available (germanium), the higher the full-energy peak efficiency.
2. The smaller the distance between the detector and the source material, the higher the full-energy peak efficiency.
3. While better resolution gives a better MDA, the resolution (like the background) contributes only as the square root to the MDA value, whereas the MDA is proportional to the full-energy peak efficiency.

Within these limits:

1. The IEEE efficiency is not a good predictor of the performance of a detector in a different detector-sample geometry.
2. When counting flat sources or filters, the detector diameter does not add much efficiency when it is more than 1.3 times the sample diameter. A longer crystal will have a higher peak-to-Compton ratio and thus a lower MDA for sample related Compton background at intermediate energies. The extra length, although it increases the cosmic background component slightly, also gives high energy efficiency.
3. The Marinelli beaker inside diameter should be as small as possible to fit over the detector endcap. For activities normalized to unit weight, a larger beaker will give lower detection limits per unit sample weight.
4. The crystal length should be long enough to have reasonable efficiency for the highest energy gamma rays of interest. Additionally, for Marinelli beaker geometry, the length should be greater than the inside diameter of the beaker.
5. There is variation from detector to detector with similar specifications. This variation is even greater when only one specification is considered. For example, there is a large combination of detector diameters and lengths that will produce the same IEEE efficiency. In characterizing these detectors for other geometries and energies, the most influential parameter may be detector diameter.

Electronics

Up until 1996, the traditional setup of electronics included a bias supply, spectroscopy amplifier, ADC, and a computer interface. ORTEC's MultiChannel Buffer, or MCB, was introduced in 1985 to provide both the computer interface and ADC functionality in a single module. A "traditional" analog set up includes the following ORTEC hardware:

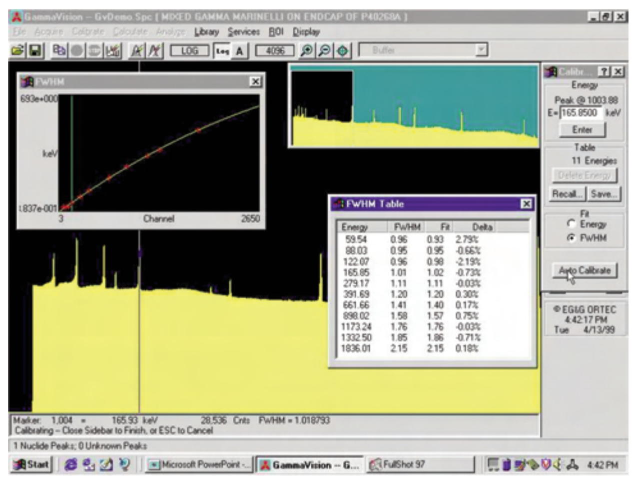
- Model 659 HPGe Bias Supply with HV shutdown protection circuit (in case of inadvertent detector warm-up)
- Model 672 Spectroscopy Amplifier
- Model 919E 4-input MCB with Ethernet interface
- 4001C/4002D NIM Bin and power supply

In 1996, ORTEC advanced the spectroscopy world by introducing the first fully integrated spectrometer based on Digital Signal Processing (DSP) technology. The DSP technology replaces the analog-shaping amplifier with a programmable digital filter. The advantages of the digital filter are: (1) more wide ranges of filter settings to better tune the electronics to the specific detector; (2) greater dynamic capability for a given group of settings for better performance at both high and low count rates; and (3) greater stability versus changes in temperatures, which reduces drift in peak position prevalent in analog shaping amplifiers.

The ORTEC DSPEC system includes the DSP technology with a built-in bias supply and Ethernet connection for computer interface. In 1999, ORTEC followed the DSPEC with the DSPEC-PLUS, which provides an even greater number of filter settings to achieve unparalleled throughput capability in high rate counting systems. Today, nearly all counting laboratories benefit from the use of DSP-based spectrometers in terms of performance, stability, reliability, and productivity.

Software Solutions — GammaVision-32 Gamma-Spectrometry Analysis Software

Windows-based spectral analysis and reporting software



GammaVision-32 is an integrated MultiChannel Analyzer (MCA) emulator and gamma spectrum analysis program for the Windows operating environment. The advanced MCA emulation and interactive analysis give complete control over the collection and analysis of germanium detector gamma-ray spectra. GammaVision incorporates the latest advances in analytical accuracy, user friendliness, and the widest range of tools and corrections available to the spectroscopist. Automation is accomplished with many features including automated sequences or "job streams." A minimum of input information is required for GammaVision-32 to produce a result. After analysis, the spectroscopist can evaluate the results using the expanded report options or a variety of on-screen, informative plotting routines.

The built-in quality assurance (QA) features monitor system performance and store the results in a Microsoft Access database. GammaVision-32 is designed to analyze spectra generated by ORTEC DSPEC hardware directly from the spectrum on display or from spectrum files on disk. GammaVision-32 uses the advanced .SPC file structure format for storage of all parameters (hardware and software) necessary to produce analysis results including efficiency, energy/resolution calibration, library, decay information, etc.

GammaVision-32 provides extensive menus and toolbars for the operation of all acquisition and analysis functions such as Start/Stop/Clear, setting of analysis parameters, calibration, and library editing. Real-time graphical displays of the spectrum and calculated peak information offer the user instant feedback on the data acquisition progress.

MCA Emulation

An MCA, in its most basic form, is an instrument that sorts and counts events in real time. This sorting is based on certain characteristic of these events, and the events are grouped together into bins for counting purposes called channels. The most common type of multichannel analysis, and the one of greatest interest to nuclear spectroscopists, is pulse-height analysis (PHA).

PHA events are signal pulses originating from a detector, and the characteristic of interest is the pulse height or voltage, which is proportional to the particle or photon energy. An analog-to-digital converter (ADC) is used to convert each pulse into a channel number, so that each channel corresponds to a narrow range of pulse heights or voltages. As pulses arrive over time, the MCA will collect in memory a distribution of the count of pulses with respect to pulse height (a series of memory locations, corresponding to ADC channels, will contain the count of pulses of similar, although not necessarily identical, height). This distribution, arranged in order of ascending energies, is commonly referred to as a spectrum. To be useful, the acquired spectrum must be available for storage and/or analysis, and is displayed on a graph whose horizontal axis represents the height of the pulse and whose vertical axis represents the number of pulses at that height, also referred to as a histogram. GammaVision-32 emulates an MCA with remarkable power and flexibility when combined with multichannel buffer (MCB) hardware (detector interface) and a personal computer. The ORTEC MCB is a unique combination of ADC and computer interface. The MCB performs the actual pulse-height analysis and stores the histogrammed spectral data with battery backed-up memory, while the computer and operating system make available the display facility, data-archiving hardware, and analysis capabilities.

The GammaVision software is the vital link that marries these components to provide meaningful access to the MCB via the user interface provided by the computer hardware. The GammaVision MCA emulation displays continuously the currently acquiring spectrum, the current operating conditions, and the available menus. All important operations that need to be performed on a spectrum, such as peak location, insertion of regions of interest (ROIs), display scaling, and sizing are implemented using either the keyboard (accelerators) or the mouse (menus and toolbars). Spectrum peak searching, report generation, printing, archiving, calibration, and other analysis tools are available from the drop-down menus. The result is an integrated analysis environment that both new and experienced users will find easy to use.

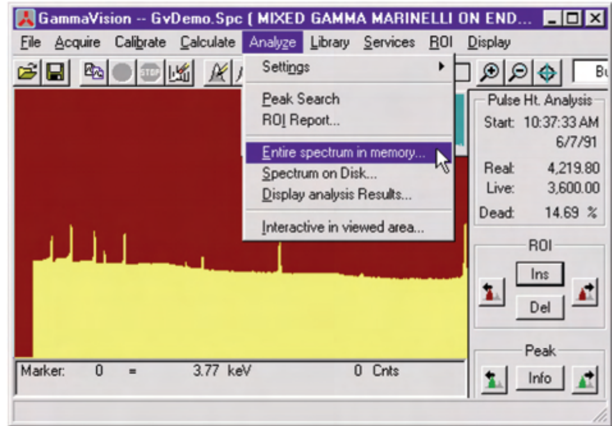
A buffer is maintained in the computer memory, to which one spectrum can be moved for display and analysis, either from detector memory or from disk, while another spectrum is collected in the detector. As much as possible, the buffer duplicates in memory the functions of the detector hardware on which a particular spectrum was collected. Data can also be analyzed directly in the detector hardware memory, as well as stored directly from the detector to disk.

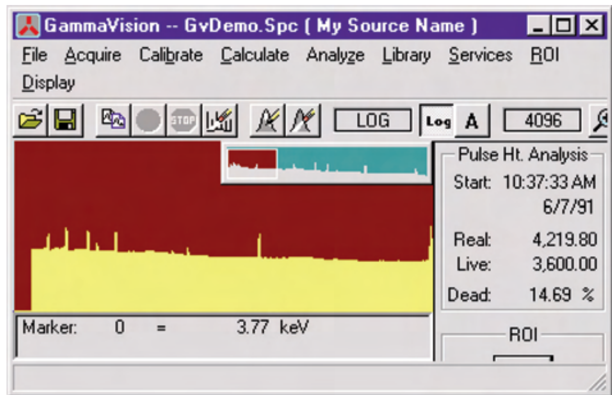
System requirements and electronic components

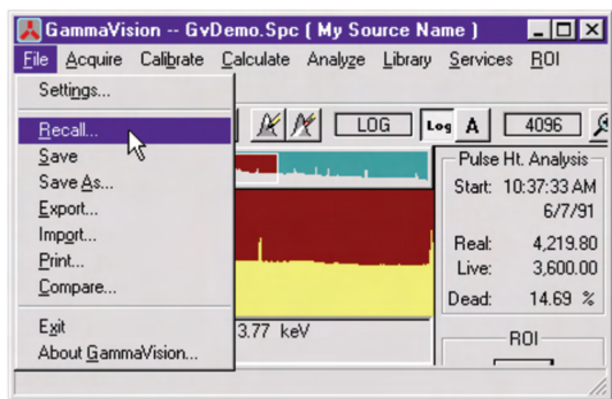
GammaVision-32 (A66-B32) will run on any IBM-compatible computer that supports Microsoft Windows 2000/XP/Vista/7. Data can be saved or retrieved from any number of disk or fixed drives. Front-end acquisition hardware supported includes ORTEC DSPEC MCBs.

GammaVision-32 can control and display up to 250 detectors, either local or networked.

GammaVision will correctly display and store a mixture of different size spectra. The detectors in any given system are assigned unique identification and have user assigned names. Expanding the system for more detectors is easy.







Environmental Gamma-Spectrometry System Summary

High-purity high-resolution Ge detectors are required for environmental gamma-ray measurements. A complete system includes a shielded detector, signal amplification and sorting devices, and data display and analysis software. Table 1 summarizes the ORTEC recommended components for a total solutions system for this application.

Table 1. Recommended components for a gamma-ray spectroscopy system using the latest technology from ORTEC – Marinelli (water sample) geometry.		
Item	Model	Description/Function
GammaVision-32	A66-B32	Gamma-spectroscopy analysis software
Ge detector (40% to 150%+)	See ORTEC catalog	High-resolution gamma-ray detector
Cryostat (low background or xlb*)	Horizontal, Vertical, J-type available	Cooling unit and housing for the detector
30 Liter Dewar	DWR-30	Liquid nitrogen (LN2) holding tank
Lead (Pb) shielding	HPLBS1 or HPLBSF1	Background suppression includes tin/copper liner
Digital Spectrometer	DSPEC Pro	Digital spectrometer and MCA (contains HV bias supply for detector and USB Computer interface)
Interface	USB	USB cable direct to PC.
Computer	Various	Any PC capable of running Windows 2000/XP/Vista/7.
*includes lead-shielded preamplifier		

Reference

(1) American National Standard for Calibration and Use of Germanium Spectrometers for Measurement of Gamma-Ray Emission Rates of Radionuclides, ANSI N42.14. (1999), IEEE, Inc., NY, NY.

(2) Gilmore, G. & Hemingway, J. D. (1995). Practical Gamma Spectrometry, John Wiley and Sons, NY, NY.

(3) American National Standard – Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control, ANSI N42.22. (1995), IEEE, Inc., NY, NY.

(4) Paulus, T. J., Keyser, R. M. Enhancement of Peal-To-Total Ratio in Gamma- Spectroscopy. Technical Note, ORTEC, Oak Ridge, TN, USA.

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(6) Keyser, R. M. Characterization and Applicability of Low-Background Germanium Detectors, Technical Note, ORTEC, Oak Ridge, TN, USA.

(7) Simpson, M. L., Raudorf, T. W., Paulus, T. J. & Trammel, R. C. (1988). Charge Trapping in Ge Spectrometers. IEEE Transactions on Nuclear Science, Nuclear Science Symposium, Orlando Florida, Vol. 36, No. 1, Nov. 1988, 260-266.

(8) Raudorf, T. W. & Pehl, R. H. (1987). Effect of Charge Trapping on Germanium Coaxial Detector Line Shapes. Nuclear Instruments and Methods in Physics Research, Vol. A255, 538-551.

Specifications subject to change
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